Reduction of threading dislocation density in SiGe layers on Si (001) using a two-step strain–relaxation procedure

Akira Sakai,[a] Ken Sugimoto, Takeo Yamamoto, Masahisa Okada, Hiroya Ikeda, and Yukio Yasuda
Department of Crystalline Materials Science, Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

Shigeaki Zaima
Center for Cooperative Research in Advanced Science and Technology, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

(Received 5 June 2001; accepted for publication 10 September 2001)

A method to obtain high-quality strain–relaxed SiGe buffer layers on Si(001) substrates is presented. In this method, the strain relaxation of the SiGe layer is performed using a two-step procedure. Firstly, a low-temperature-grown SiGe layer, whose surface is covered by a thin Si cap layer, is thermally annealed. At this stage, the strain is incompletely relaxed and an atomically flat surface can be realized. Then, a second SiGe layer is grown on the first layer to achieve further strain relaxation. Transmission electron microscopy has clearly revealed that dislocations are dispersively introduced into the first SiGe/Si substrate interface and thus no pile up of dislocations occurs. The formation of a periodic undulation on the growth surface of the second SiGe layer is the key to inducing a drastic reduction in the threading dislocation density. [DOI: [10.1063/1.1419037]

The application of SiGe/Si heterostructures to Si-based integrated semiconductor devices allows the possibility for improvement in their transport properties. In particular, devices with a strained Si channel are promising candidates for the realization of mobility enhancement and, therefore, have been extensively studied with regard to their feasibility in advanced complementary metal–oxide–semiconductor devices.[1] One of the prominent issues to be addressed for the introduction of such heterostructures into a production environment is the quality of the strain–relaxed SiGe buffer layer which is used as a virtual substrate to induce in-plane tensile strain into the Si channel. Since the structural properties of the buffer layer directly influence the channel carrier transport characteristics of the device, the buffer layer needs to have an atomically flat surface and a low threading dislocation density. It must be also fully strain relaxed to allow precise strain tuning of the channel. Although compositionally graded layers (CGL) have been successfully applied to various types of devices,[2–4] these still have some disadvantages. For example, CGLs often exhibit surface roughness in the form of a so-called cross hatch pattern, which is closely related to the pile up of misfit dislocations introduced into the layer via the specific stain–relaxation mechanism.[5–7] Furthermore, the CGL is conventionally required to be several μm in thickness, a value which is too thick for the practical monolithic integration of devices. This may become a serious drawback in the time-intensive device fabrication process. Therefore, there exists a number of problems to be solved for the implementation of relaxed SiGe buffer layers to integrated devices.

In this work, we have taken an approach to obtain high-quality strain–relaxed SiGe buffer layers on Si(001). Our method consists of two strain–relaxation stages; the first of which is the annealing of a pseudomorphic SiGe layer with a cap layer and the second is the subsequent growth of SiGe on that layer. This procedure greatly suppresses the pile up of misfit dislocations and creates a characteristic dislocation morphology in which dislocations dispersively exist at the first SiGe/Si substrate interface. A considerable reduction of threading dislocation density is also verified through transmission electron microscopy (TEM).

Growth of SiGe layers was carried out using a solid-source molecular-beam epitaxy system (MBE) whose base pressure was less than 1×10^{-10} Torr. After the cleaning of a Si(001) substrate surface, a $Si_{0.7}Ge_{0.3}$ layer with a thickness in the range of 50 to 100 nm was grown on the substrate at a substrate temperature of 400 °C, which is lower than that used in the conventional MBE procedure. Although the thickness range used here is beyond the critical thickness predicted by the Matthews–Blakeslee theory,[8] the misfit dislocations are kinetically inhibited due to the low growth temperature and, as discussed later, no strain-relaxation was actually observed in these samples. Reflection high-energy electron diffraction (RHEED) patterns also showed 1/8 order diffraction streaks reflecting a (2×8) reconstruction, which is typical of a strained SiGe film. Next, a thin Si cap layer with a thickness of 5 nm was grown on the $Si_{0.7}Ge_{0.3}$ layer at the same temperature before annealing. The thin cap layer effectively suppresses surface roughening during the annealing due to a reduction of the surface stress of the film. A detailed mechanism has been described elsewhere.[9,10] The sample was then annealed at a temperature ranging from 600 °C to 800 °C for 5 min as the first procedure for strain relaxation. For the second stage of strain relaxation, a $Si_{0.7}Ge_{0.3}$ layer with a thickness ranging from 50 to 400 nm was grown on the first layer at 600 °C.

[a]Electronic mail: sakai@alice.xtal.nagoya-u.ac.jp

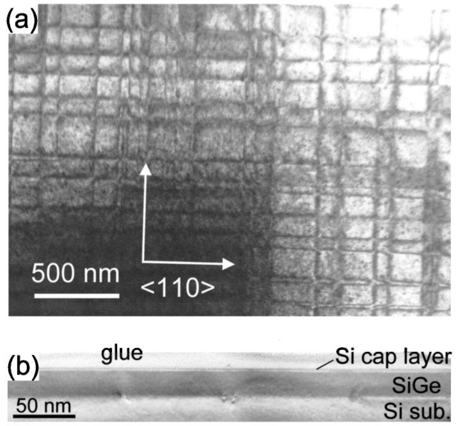

FIG. 1. (a) Plan-view and (b) cross sectional TEM images of the 600 °C-annealed sample having the following structure; Si cap layer (5 nm)/$Si_{0.7}Ge_{0.3}$ layer (50 nm)/Si(001).

First, we have checked the effect of the cap layer on suppressing the surface roughening of the SiGe layers. *In situ* RHEED observations were performed on the surfaces of the first SiGe layers with and without the cap layer subjected to annealing. A drastic difference was observed; the surface with the cap layer showed a streaky pattern characteristic of a flat surface morphology, while that without the cap layer showed a spotty pattern resulting from three-dimensional clustering of SiGe. This fact is consistent with the previous results[10] and unambiguously indicates that the cap layer explicitly suppresses the surface roughening. By unloading the samples from the chamber and by using atomic force microscopy (AFM), we measured the root-mean-square roughness and obtained a value of 0.67 nm for $5\times5\ \mu m^2$ scan for the 600 °C-annealed sample with the cap layer.

Figure 1 shows the results of a TEM observation of the sample with the cap layer just after annealing of 600 °C. In the plan-view TEM image shown in Fig. 1(a), it can be clearly seen that dislocations run along two orthogonal $\langle 110\rangle$ directions. These dislocations were found to lie at the SiGe/Si substrate interface, as shown by the cross sectional TEM image of Fig. 1(b), where the flat surface is consistent with the AFM results. The characters of the dislocations were determined by conventional diffraction analysis and by direct imaging of their core structures.[10] As a result, we found that most of dislocations observed here had the characters of 60° dislocations with a Burgers vector of the $\mathbf{a}/2\langle 110\rangle$ type which is inclined with respect to the interface. The mean separation between individual dislocations was estimated to be 100 nm. From this value and the Burgers vector component effective in relaxing the in-plane compressive strain, we find that about 20% of the compressive strain in the SiGe layer can be relaxed in this initial stage of strain relaxation.

Double crystal X-ray diffraction (XRD) of Si(004) and SiGe(004) reflections was carried out for capped SiGe layers prepared under various conditions. As shown in Fig. 2, the SiGe(004) Bragg peak in the sample with the 50 nm-thick first SiGe layer before the 600 °C annealing coincided with the completely strained $Si_{0.7}Ge_{0.3}$ peak position. After the annealing, the peak shifted to a higher angle position, due to the onset of strain relaxation. The value of $23\pm1\%$ relaxation estimated from the shift is in good agreement with the

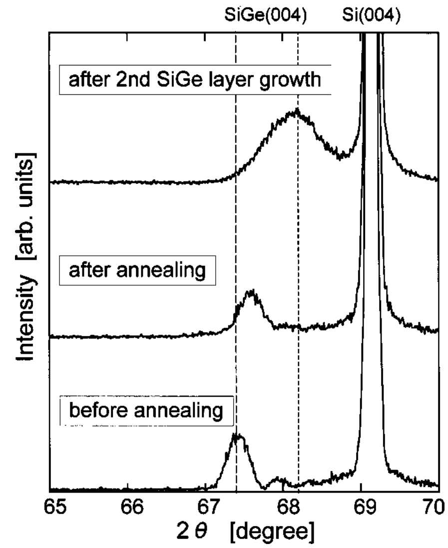

FIG. 2. XRD peak profiles for SiGe(004) and Si(004) reflections in samples before and after annealing at 600 °C and a sample after the second SiGe growth. These samples had 50 nm-thick first $Si_{0.7}Ge_{0.3}$ layers with a 5 nm-thick Si cap layer. The thickness of the second layer was 200 nm. The broken (dotted) line indicates the ideal peak position for a completely strained (100% strain–relaxed) $Si_{0.7}Ge_{0.3}$ film.

value from the misfit dislocation density evaluation discussed. The peak shift at this stage was found not to be so sensitive to the annealing temperature. We observed no additional peak shift in samples with the same thickness of SiGe when the temperature was increased up to 800 °C. This suggests that during the 600 °C anneal, the first layer almost reaches the misfit dislocation density which is determined by the net stress resulting from a balance between the thickness of the strained layer and the self-energy of the dislocation created. In fact, we have confirmed the achievement of about 70% strain relaxation in samples with 100 nm-thick initial layers after annealing at 600 °C.

A remarkable degree of strain relaxation was completed after the second $Si_{0.7}Ge_{0.3}$ layer growth. In the XRD profile shown in Fig. 2, the peak shift corresponding to a $92\pm1\%$ strain relaxation was clearly observed. We have also examined defect structures in SiGe after the second layer growth. Figure 3(a) is a representative cross sectional TEM image of a sample having a $Si_{0.7}Ge_{0.3}$(200 nm)/Si-cap (5 nm)/$Si_{0.7}Ge_{0.3}$(50 nm)/Si(001) structure. A smooth interface with no dislocations is clearly observed between the

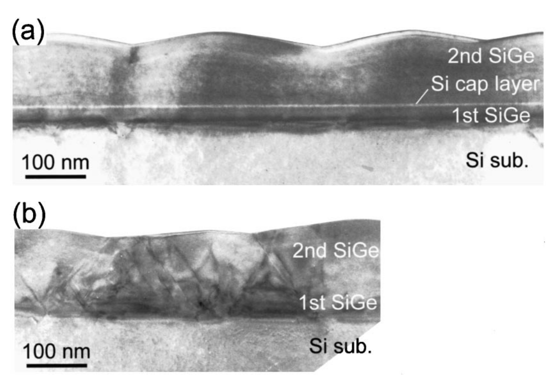

FIG. 3. Cross sectional TEM images of samples after the growth of a 200 nm-thick second SiGe layer (a) with and (b) without a Si cap layer. Note the difference between the threading dislocation densities in the two samples.

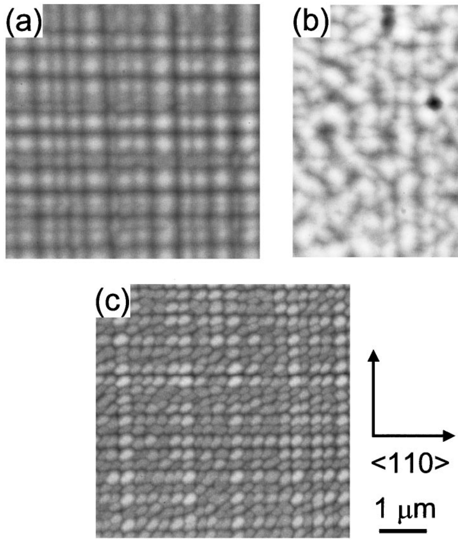

FIG. 4. AFM images of samples after the growth of the second SiGe layer. The second layers of 200 nm thickness were grown (a) with a Si cap layer, (b) without a Si cap layer. (c) The second layer of 50 nm thickness was grown with a Si cap layer.

second SiGe layer and the cap layer. Note that threading dislocations are almost absent from the observed area. We find that almost all misfit dislocations are confined at the first SiGe/Si substrate interface. These dislocations tend to be dispersed at the interface and the pile up of the dislocations, which is often observed in CGL, is hardly observed. Using plan-view TEM observations, the threading dislocation densities in these samples were estimated to be on the order of $10^7\ \mathrm{cm}^{-2}$. For comparison, we have also observed SiGe layers without the cap layer; the samples were grown by a two-step procedure in which low-temperature-grown $Si_{0.7}Ge_{0.3}$ layers were annealed at 600 °C without forming the cap layer and followed by a 200 nm-thick $Si_{0.7}Ge_{0.3}$ layer growth. Figure 3(b) shows a typical defect structure with many threading dislocations observable in the SiGe layer. The threading dislocation density was estimated to be more than $10^{10}\ \mathrm{cm}^{-2}$. These results unambiguously indicate that the cap layer has an effect in reducing the threading dislocation density after the growth of the second SiGe layer.

It is evident from the aforementioned results that considerable strain relaxation occurs during the second SiGe layer growth and that this relies on the misfit dislocations introduced into the first SiGe/Si substrate interface. At this growth stage, the cap layer plays a crucial role in decreasing the number of threading segments from the misfit dislocations and avoiding pile up of the dislocations. In order to reveal this effect of the cap layer, we focus on the interplay between surface morphological changes and dislocation introduction. In Fig. 3(a), an undulation with a wavelength of about 300 nm can be also seen on the surface. Figure 4(a) is an AFM image showing the corresponding surface of the second SiGe layer. We find a quasiperiodic surface undulation, in contrast to the case without the cap layer shown in Fig. 4(b). The AFM image of Fig. 4(c) shows the surface of a layer at an earlier stage during the growth of the second SiGe layer on top of the cap layer. In this sample, SiGe islands are observed which were found aligned along rows parallel to the orthogonal ⟨110⟩ directions. Similar to the case of Xie *et al.*,[11] the incompletely relaxed first SiGe layer with the cap layer produces a regularly distributed variation in the in-plane lattice constant on the surface due to the strain field associated with the misfit dislocation network buried at the interface. Since SiGe islands preferentially nucleate over the misfit dislocation network, they arrange in an ordered form, resulting in a surface undulation that conforms to the orthogonal ⟨110⟩ directions after the subsequent growth.

We deduce that this undulation determines the generation and propagation behavior of dislocations during the growth of the second SiGe layer. By noting that a cusp in the surface undulation acts as a preferential nucleation site for misfit dislocations,[12] it is likely that dislocations are simultaneously introduced at every cusp on the surface to relax the strain. In other words, the strain relaxation is dominated by the introduction of new dislocations from the surface rather than the multiplication of the existing interfacial dislocations via, for example, a modified Frank–Read mechanism.[6] Thus, the pile up of dislocations is effectively avoided in our case. Furthermore, the periodicity of the undulation creates a regular strain field in the SiGe layer. This greatly enhances the possibility of dislocation propagation along the ⟨110⟩ directions for long distances, so that the threading segments then have the opportunity to travel the entire length of the sample. Consequently, pinning of threading dislocations should be considerably suppressed. By contrast, in the case of the sample without the cap layer, surface roughening takes place at the first stage of the strain relaxation. This induces a random generation of misfit dislocations, resulting in an increased density of threading segments.

In summary, we have grown strain–relaxed SiGe layers on Si(001) substrates using a two-step strain–relaxation procedure. The presence of a cap layer is the key to not only suppressing the surface roughening of the first SiGe layer during the strain relaxation process, but also to forming a periodic undulation in the second SiGe layer surface. This undulation plays an important role in decreasing the number of threading segments of misfit dislocations and avoiding the pile up of dislocations.

[1] For example, J. J. Welser, J. L. Hoyt, S. Takagi, and J. F. Gibbons, Tech. Dig. - Int. Electron Devices Meet. **94**, 373 (1994); T. Mizuno, S. Takagi, N. Sugiyama, J. Koga, T. Tezuka, K. Usuda, T. Hatakeyama, A. Kurobe, and A. Toriumi, Tech. Dig. - Int. Electron Devices Meet. **99**, 934 (1999).
[2] D. K. Nayak, J. C. S. Woo, J. S. Park, K. L. Wang, and K. P. MacWilliams, Appl. Phys. Lett. **62**, 2853 (1993).
[3] K. Ismail, M. Arafa, K. L. Saenger, J. O. Chu, and B. S. Meyerson, Appl. Phys. Lett. **66**, 1077 (1995).
[4] N. Sugii, K. Nakagawa, S. Yamaguchi, and M. Miyao, Appl. Phys. Lett. **75**, 2948 (1999).
[5] B. S. Meyerson, K. L. Uram, and F. K. LeGoues, Appl. Phys. Lett. **53**, 2555 (1988).
[6] F. K. LeGoues, B. S. Meyerson, J. F. Morar, and P. D. Kirshiner, J. Appl. Phys. **71**, 4230 (1992).
[7] M. A. Lutz, R. M. Feenstra, F. K. LeGoues, P. M. Mooney, and J. O. Chu, Appl. Phys. Lett. **66**, 724 (1995).
[8] J. W. Matthews and A. E. Blackeslee, J. Cryst. Growth **27**, 118 (1974); *ibid.* **29**, 273 (1975); *ibid.* **32**, 265 (1976).
[9] N. Ikarashi and T. Tatsumi, Jpn. J. Appl. Phys., Part 2 **36**, L377 (1997).
[10] A. Sakai, T. Tatsumi, and K. Aoyama, Appl. Phys. Lett. **71**, 3510 (1997).
[11] Y. H. Xie, S. B. Samavedam, M. Buisara, T. A. Langro, and E. A. Fitzgerald, Appl. Phys. Lett. **71**, 3567 (1997).
[12] D. E. Jesson, S. J. Pennycook, J.-M. Baribeau, and D. C. Houghton, Phys. Rev. Lett. **71**, 1744 (1993).